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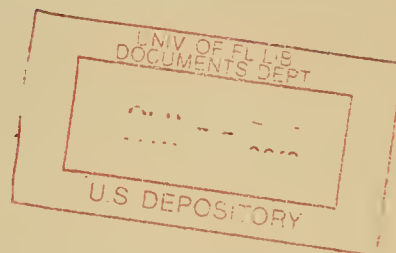
CHEMICAL PROBLEMS IN STABLE ISOTOPE SEPARATION

by

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Tennessee Eastman Corporation

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CHEMICAL PROBLEMS IN STABLE ISOTOPE SEPARATION

By Arthur J. Miller and Boyd S. Weaver

The Development Division of the Clinton Engineer Works-Tennessee Eastman Corporation has for more than a year utilized the electromagnetic method for separating the naturally occurring isotopes of elements other than uranium. In this program the Division's Chemical Laboratories have been engaged in preparing charge materials for the electromagnetic units, recovering, chemically purifying, and analyzing the separated isotopes, chemically reconditioning the units, and preparing mass spectrometer charges from the isotope concentrates for determinations of isotopic abundance. To date, 19 elements have been processed and concentrates of 63 different isotopes made available to the nuclear physicists and chemists of the Manhattan Project. Most of these isotopes are stable or not radioactive. The concentrates are listed in Table 1. Isotopes of several of the 19 elements and those of silicon, zirconium and antimony are in various stages of chemical purification.

In dealing with charge materials for the electromagnetic separators, there are several factors taken into account. Of considerable importance is the question as to whether the use of a given material will supply fundamental information concerning electromagnetic operations. Next, for some elements, it must be decided whether it would be advisable to prepare, at considerable expense, compounds capable of satisfactory performance in existing unit types or whether to modify equipment. Problems in chemical reconditioning of the units such as toxicity of the charge, explosion hazards, and corrosion of the units must also be considered.

Regardless of how these decisions are made, the charge material chosen is supplied conforming as closely as possible to the following specifications:

- 1) That it can be volatilized at a controlled rate.
- 2) That it contains a minimum of impurities that will volatilize.
- 3) That it contains a minimum of impurities that will ionize.
- 4) That it contains a minimum of impurities whose isotopes are isobaric with those to be collected.

To recover isotope concentrates as more or less pure chemical compounds of known composition is an exercise in chemical ingenuity for each element. The problem generally resolves itself into one of economically isolating minute quantities of the isotope from large amounts of other materials. The extent of recovery is usually determined by balancing the cost of charge material and isotope separation against recovery cost. For example, if 99.9% can be obtained with one-tenth the effort of recovering the remaining 0.1% the processing will likely cease at such a point. Therefore, it is the continuous responsibility of the chemists to devise low cost methods which provide a maximum of recovery.

The chemical purity achieved is often influenced by the end usage planned for the individual concentrate. One requirement always considered is the necessity of minimizing impurities that contain isobars of the element in order to obtain a satisfactory isotopic analysis in the mass spectrometer. Typical spectrographic chemical analyses are shown in Table 2.

The mass spectrometer charge usually consists of a portion of the isotope concentrate converted to a compound suitable for spectrometer operation. To carry out the synthesis without loss of valuable

material or contamination with undesirable impurities is another major chemical problem with each element encountered.

Interest in most of the stable isotopes is still mainly confined to the field of nuclear research. However, simplification and increased availability of methods for determining isotopic abundance would make them invaluable as tracers in chemical experimentation.

The future program as outlined by Dr. C. E. Larson, Director of the Development Division, calls for the eventual concentration of the isotopes of additional elements including those of the rare earths.

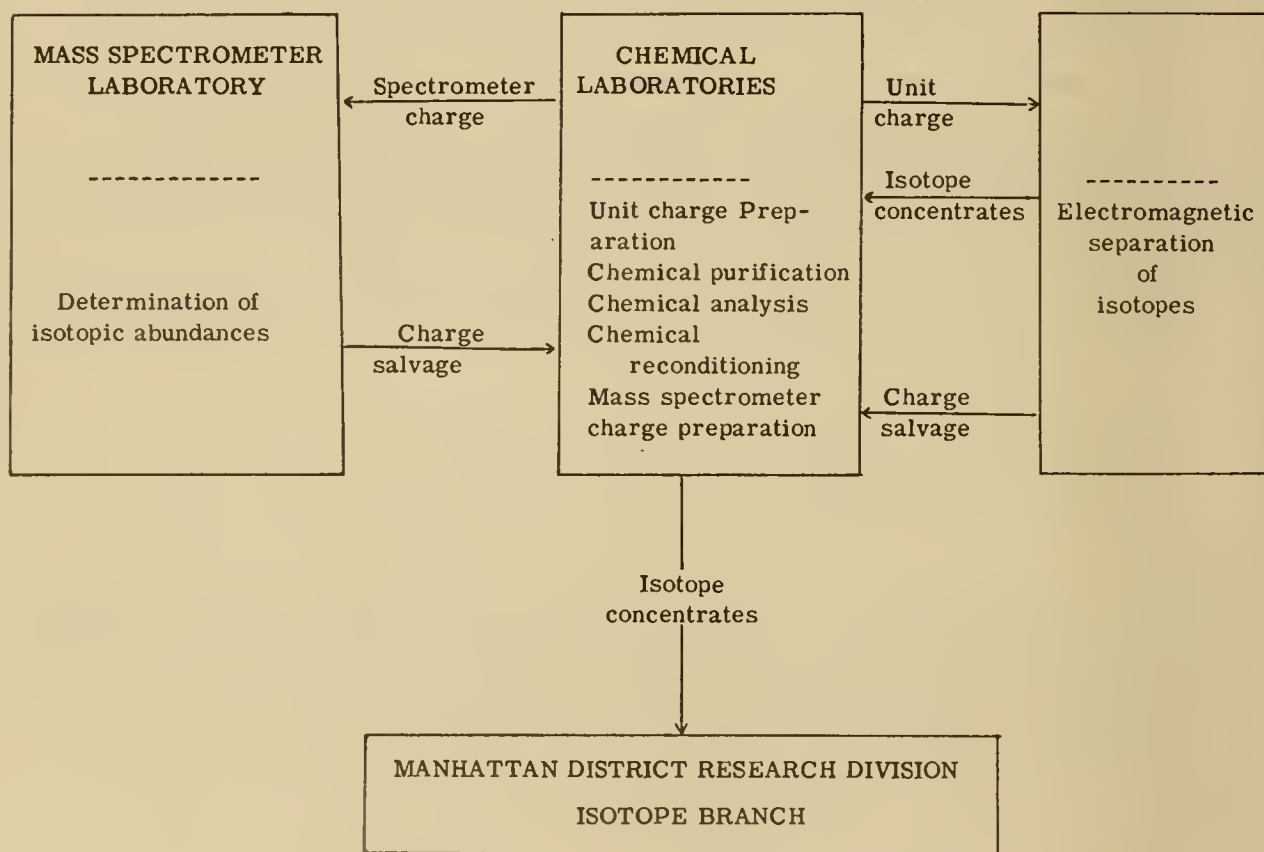


Figure 1. Material flow in isotope separation.

Table 1. Isotopic concentrates.

Element	Isotope	Abundance in concentrate (%)	Natural abundance (%)
Li	6	93.91	7.5
Li	6	87.76	7.5
Li	7	99.89	92.5
Li	7	99.91	92.5
C	12	*	98.9
C	13	*	1.1
Mg	24	*	77.4
Mg	24	*	77.4
Mg	25	*	11.5
Mg	25	*	11.5
Mg	25	*	11.5
Mg	26	*	11.1
Mg	26	*	11.1
Mg	26	*	11.1
K	39	99.93	93.38
K	40	0.16	0.012
K	41	88.36	6.61
Ca	40	99.95	96.96
Ca	40	99.97	96.96
Ca	43	*	0.15
Ca	44	*	2.06
Cr	50	73.76	4.49
Cr	52	98.80	83.76
Cr	53	88.59	9.43
Cr	54	61.0	2.30
Fe	54	55.47	6.04
Fe	54	42.85	6.04
Fe	54	81.06	6.04
Fe	54	83.03	6.04
Fe	56	98.41	91.57
Fe	56	98.70	91.57
Fe	56	98.62	91.57
Fe	56	97.42	91.57
Fe	57	21.10	2.11
Fe	57	56.13	2.11
Fe	57	73.01	2.11
Fe	57	69.63	2.11
Fe	58	22.0	0.28
Fe	58	*	0.28
Fe	58	*	0.28
Ni	58	98.05	67.4
Ni	58	98.51	67.4
Ni	60	94.40	26.7
Ni	60	87.10	26.7

Table 1. Isotopic concentrates. (Continued)

Element	Isotope	Abundance in concentrate (%)	Natural abundance (%)
Ni	61	78.83	1.2
Ni	61	34.42	1.2
Ni	62	94.25	3.8
Ni	62	67.99	3.8
Ni	64	85.10	0.88
Ni	64	64.20	0.88
Cu	63	97.0	70.13
Cu	63	99.35	70.13
Cu	65	93.81	29.87
Zn	64	*	50.9
Zn	66	*	27.3
Zn	67	*	3.9
Zn	68	*	17.4
Zn	70	*	0.5
Se	80	*	48.0
Br	79	*	50.6
Br	81	*	49.4
Sr	84	*	0.56
Sr	86	*	9.86
Sr	88	*	82.56
Mo	92	92.07	22.0
Mo	94	74.68	9.4
Mo	95	*	16.1
Mo	96	*	16.6
Mo	97	*	9.65
Mo	98	*	24.1
Mo	100	*	9.25
Mo	100	*	9.25
Ag	107	88.96	51.9
Ag	107	90.26	51.9
Ag	109	*	48.1
Ag	109	92.16	48.1
Cd	106	*	1.4
Cd	111	*	13.0
Cd	113	*	12.3
Cd	114	*	28.0
Cd	116	*	7.3
In	113	*	4.5
In	115	99.56	95.5
Sn	112	*	1.1
Sn	122	*	5.5
Sn	124	*	6.8

Table 1. Isotopic concentrates. (Continued)

Element	Isotope	Abundance in concentrate (%)	Natural abundance (%)
Pb	204	7.8	6.7
Pb	206	75.67	23.6
Pb	207	61.55	22.6
Pb	208	92.10	52.3

* Isotopic abundance not obtained to date.

Table 2. Chemical purity of typical concentrates.

Impurity	Mg ²⁶	Ca ⁴⁰	Cu ⁶³	Cu ⁶⁵	Zn ⁶⁴	Cd ¹¹⁴	In ¹¹⁵
Ag	N.D.	N.D.	0.04%	0.06%	< 0.04%	N.D.	< 0.04%
Al	< 0.04%	N.D.	N.D.	N.D.	N.D.	N.D.	0.08%
As	N.D.	---	N.D.	N.D.	N.D.	---	---
B	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Ba	---	N.D.	---	---	---	---	---
Be	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Ca	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Cd	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Co	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Cr	N.D.	N.D.	N.D.	N.D.	< 0.15%	N.D.	N.D.
Cu	0.04%	N.D.			< 0.04%	< 0.04%	< 0.04%
Fe	0.08%	0.08%	< 0.04%	0.04%	N.D.	N.D.	0.08%
In	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	
K	---	N.D.	---	---	---	---	---
Li	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Mg		0.04%	< 0.02%	< 0.02%	< 0.02%	N.D.	0.02%
Mn	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	< 0.04%
Mo	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Na	< 0.08%	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Ni	N.D.	N.D.	< 0.08%	N.D.	N.D.	N.D.	N.D.
Pb	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Pt	< 0.15%	---	N.D.	N.D.	N.D.	---	---
Rb	---	N.D.	---	---	---	---	---
Si	0.05%	0.05%	< 0.05%	0.05%	< 0.05%	< 0.05%	0.31%
Sn	N.D.	N.D.	N.D.	N.D.	N.D.	< 0.08%	N.D.
Sr	---	N.D.	N.D.	N.D.	N.D.	N.D.	< 0.11%
Ti	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
V	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	< 0.08%
Zn	N.D.	N.D.	N.D.	N.D.		0.15%	N.D.
Zr	---	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

N.D. - Not detected spectrographically

< - Detected but below limit of determination

--- - Not determined

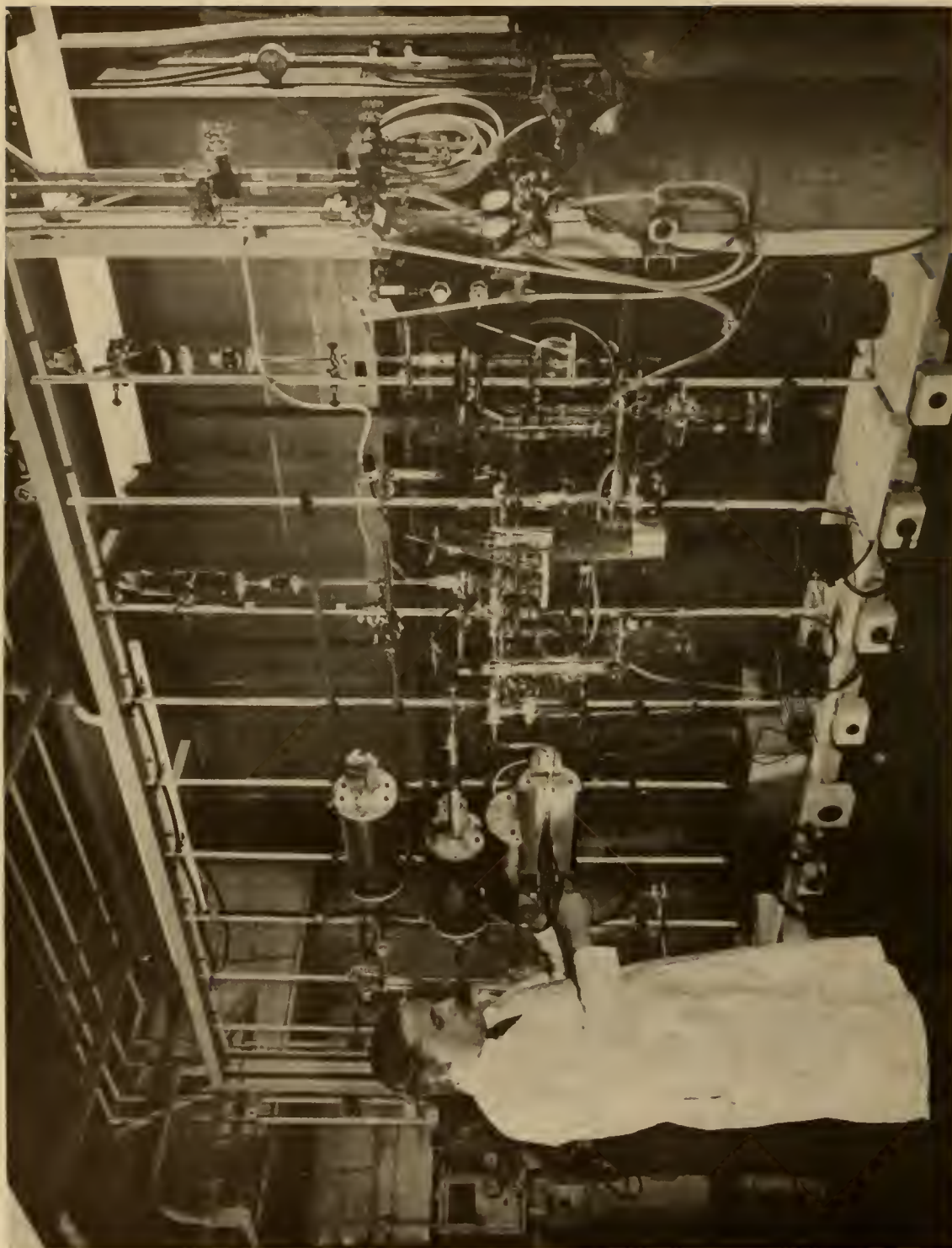


Figure 2. Preparation of spectrometer charge for Mo^{97} .

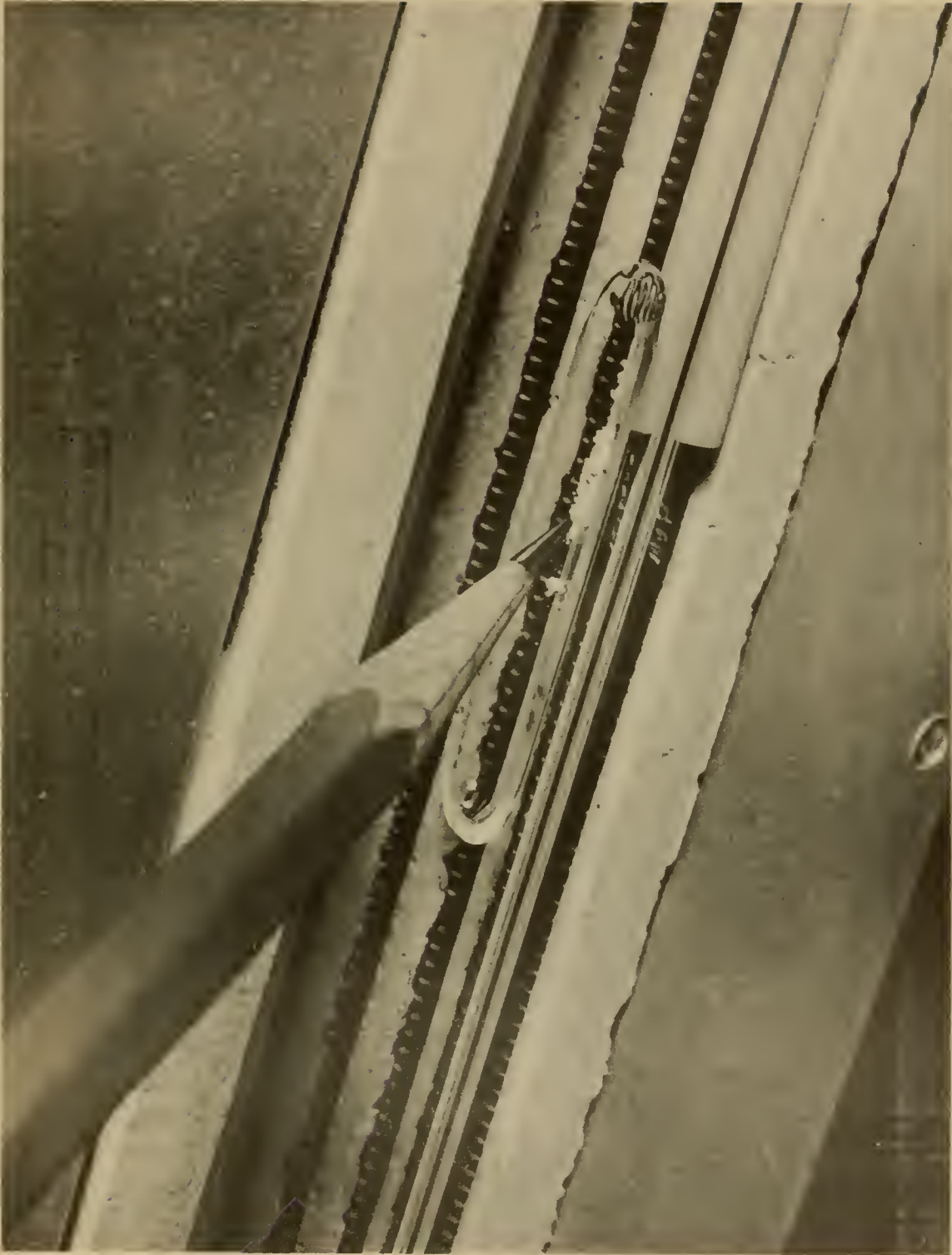
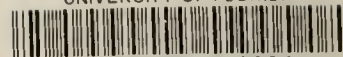


Figure 2A. Close-up of spectrometer charge preparation.



Figure 3. Close-up of Fe⁵⁸ for shipment as Fe₂O₃.

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